MULTIPYCNOMETER

Instrument Models:
MVP-6DC
MVP-D160E

OPERATING MANUAL

P/N 05034 Rev F

QUANTACHROME WARRANTY POLICY

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For the period commencing with the date of shipment and ending one year later, Quantachrome will, at its option either repair or replace any part within an instrument that is found by us to be defective in material or workmanship, without charge to the customer, at our facility or at a customer's facility if the instrument purchased is backed by Quantachrome's on-site warranty as evidenced by the sales contract.

The customer is responsible for all transportation charges to our factory.

Damages during the warranty period resulting from unstable utilities, operator error or unauthorized repairs will not be covered by this warranty.

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The following limits apply to our warranty:

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Expendable items are warranted for 90 days for other than glassware breakage. Such items include, but are not limited to, sample tubes, lamps, fuses, valve plungers, seals, o-rings & other seals, hoses, flexible tubing, thermocouple vacuum tubes, filters, oils and other fluids.

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Warranty becomes void in the event of modifications or repairs by persons other than Quantachrome's service personnel, unless permission is given in writing for such repairs or modifications. Warranty is also void in the event of exposure to corrosive atmospheres of any kind.

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CALIBRATION VALUES

LARGE CALIBRATION SPHERE	V_{cal} large = 56.5592 cm^3
MICRO CALIBRATION SPHERES (2) 1.0725 cm^3 each	V_{cal} micro = 2.145 cm^3
LARGE SAMPLE CELL VOLUME	V_c large = _____cm^3
LARGE REFERENCE VOLUME	V_{Ref} large = _____cm^3
SMALL SAMPLE CELL VOLUME	V_c small = _____cm^3
SMALL REFERENCE VOLUME	V_{Ref} small = _____cm^3
MICRO CELL VOLUME	V_c micro = _____cm^3
MICRO REFERENCE VOLUME	V_{Ref} micro = _____cm^3

Model Number _____ Serial Number _____

I. INTRODUCTION

"Pycnometer" is derived from the Greek word pyknos which has long been identified with volume measurements. The MULTIPYCNOMETER is an instrument specifically designed to measure the true volume of a variety of solid materials by employing Archimedes' principle of fluid displacement and Boyle's Law of gas expansion. The displaced fluid is a gas which can penetrate the finest pores to assure maximum accuracy. For this reason helium is recommended since its small atomic dimension assures penetration into crevices and pores approaching two Ångströms (2×10^{-10}m). Its behavior as an ideal gas is also desirable. Other gases such as nitrogen can also be used, often with no measurable difference.

It is used to determine the true volume of solid or powder samples by measuring the pressure difference when a known quantity of gas under pressure is allowed to expand from a precisely known reference volume into a sample cell holder, also of known volume, containing the sample cell with sample.

Figure 1 is a flow diagram of the MULTIPYCNOMETER. The shaded area represents the known reference volume(s) V_R. After the system is purged with analysis gas, the valve that admits gas in to the system is closed, the selector valve between V_R and the cell holder V_C is turned to connect them, and the vent valves are opened. The system is now at ambient pressure P_a and the state of the sample cell with sample is defined by

$$P_a\ (V_C\ -\ V_S) = n_a\, RT_a \tag{1}$$

where n_a is the number of moles of gas occupying the calibrated cell volume (V_C) with sample present of volume (V_s), R is the gas constant and T_a is ambient temperature in kelvin.

When the reference volume alone is pressurized above ambient (after isolating it from the sample cell holder), the state of the reference volume (V_R) can be expressed as

$$P_1 V_R = n_1\, RT_a \tag{2}$$

where P_1 represents a pressure above ambient (17 psig, ~120kPa, for example) and n_1 is the total number of moles of gas in the reference volume (V_R).

When the selector valve is turned again to connect the reference volume to the sample cell holder, the pressure will fall to a lower pressure P_2, given by

$$P_2\left(V_C - V_S + V_R\right) = n_a\, RT_a\ +\ n_1\, RT_a \tag{3}$$

Substituting into equation (3) $P_a(V_C - V_S)$ and P_1V_R for n_aRT_a and n_1RT_a, respectively, gives

$$P_2\left(V_C - V_S + V_R\right) = P_a\left(V_C - V_S\right) + P_1V_R \tag{4}$$

or

$$\left(P_2 - P_a\right)\left(V_C - V_S\right) = \left(P_1 - P_2\right)V_R \tag{5}$$

Then

$$V_C - V_S = \frac{P_1 - P_2}{P_2 - P_a}\,V_R \tag{6}$$

Since P_a is made to read zero on the digital meter, that is, all pressure measurements are relative to P_a, equation (6) becomes

$$V_C - V_S = \frac{P_1 - P_2}{P_2}\,V_R \tag{7}$$

or

$$V_S = V_C - V_R\left[\left(P_1 / P_2\right) - 1\right] \tag{8}$$

Equation (8) is the working equation employed with the MULTIPYCNOMETER.

II. INSTALLATION AND COMPONENTS

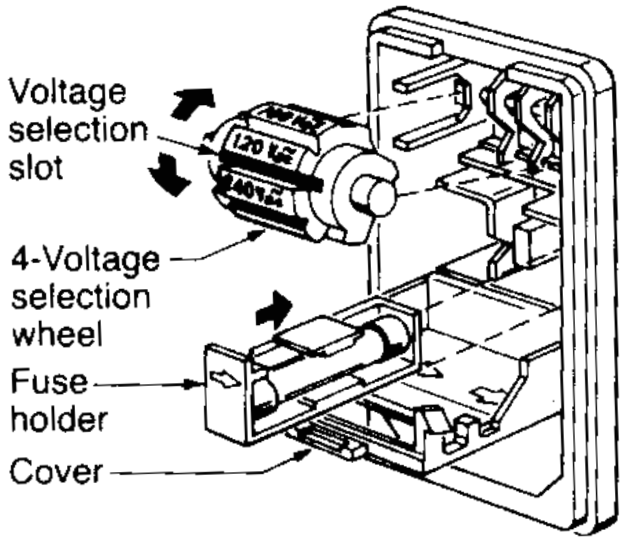
Location

Place the MULTIPYCNOMETER on a level surface away from sources of heat and cold such as radiators, air conditioning vents, direct sunlight, etc.

Mains Power

Ensure that the voltage selector at the rear of the unit matches your local mains supply. The MULTIPYCNOMETER is designed for operation on 100V, 120V, 220V or 240V, 50/60 Hz. It was shipped from the factory set for the voltage according to the purchase order which should match the voltage on the serial number plate at the rear of the unit. If the selector needs to be changed refer to the diagram below and carefully follow these steps:

1. Make sure the unit is switched OFF and unplug the power cord from its socket on the unit.
2. Pry open the plastic cover by inserting a screwdriver behind the tab on the side away from the socket.
3. Using long-nosed pliers, gently pull out the wheel located behind the plastic cover.
4. The 4 voltages are printed on the wheel. Re-insert the wheel with the voltage required facing out, towards you. Then close the plastic cover. Ensure that the voltage visible in the opening in the plastic cover matches your local mains supply before reconnecting the power cord.



Gas Tank Connections Tools required: 7/16" wrench (not supplied).

Attach a dual stage gas regulator to a cylinder of analysis gas, usually ultra-high purity helium or nitrogen. Connect the output of the regulator to the compression fitting on the right side of the cabinet using the 1/8" copper tubing and nut & ferrule set supplied.

Do not use plastic tubing. If you need to extend the gas supply line always use clean copper (or stainless steel) tubing with appropriate metal compression fittings.

It will be necessary to obtain an adapter to connect the tubing to the regulator if it does not have a 1/8" Swagelok® brand fitting. A suitable regulator assembly (P/N 01207) complete with shut-off valve, CGA580 cylinder connector and 1/8" Swagelok® brand outlet fitting is available from Quantachrome.

Adjust the pressure regulator to slightly above 20 psig (140 kPa). *Pressures above 25 psig could potentially damage the MULTIPYCNOMETER's pressure transducer.*

Check all connections for leaks using soap solution.

Sample Cells and Sample Cell Holder

Three sample cells (stainless steel cups) and two adapter sleeves are supplied with the MULTIPYCNOMETER. Sample cells are placed in the chamber with the screw cap - the *sample cell holder*. To open the sample cell holder, rotate the cap's outer ring counter-clockwise until it can be lifted off. To remove the sample cell insert the lift out tool into one of the holes near the top of the cell and lift it out of the holder.

Sample cells should be removed from the unit before being filled with sample or adding calibration spheres. Use the largest amount of sample available for best accuracy. If you do not have enough sample material to fill the large cell (135 cm^3) to at least 50% of its volume, use the small cell (20 cm^3) and the micro cell (4.5 cm^3) for even smaller quantities. The small and micro cells are always used with the appropriate adapter sleeve.

When calibrating a cell, especially the large cell, do not simply drop the large sphere into the cell but turn the cell sideways and roll the sphere into it to prevent deforming the cell.

When using the large sample cell, ensure that the horizontal slit(s) near the top of the cell line up with the vertical groove(s) machined into the cell holder. This ensures that the gas has a free path into and out of the cell.

Before replacing the cell holder cap make sure that its O-ring[1] is undamaged, clean, lightly greased[2] and is secured in the groove inside the cap. Align the fiduciary mark on the cap with the line on the cabinet ring; a locating pin on the bottom side of the cap slots into a hole in the top edge of the sample cell holder. Then rotate the outer ring of the cap clockwise (on the threads of the cell holder) until metal to metal contact is made.

1 Spare O-rings are sold in packs of two, p/n 51000-032.
2 Use Vacuum Grease P/N 91000-.25 to lubricate O-ring, never petrolatum.

Toggle Valves

Toggle valves are used to start and stop gas flow in and out of the unit (see Sections III and IV) and to select the appropriate reference volume according to the size of sample cell being used (see Section IV).

They are closed when their handles are parallel to the cabinet face and open when perpendicular to the cabinet face. Closing the toggle valves can sometimes cause a slight pressure increase in the sample cell which can be observed on the pressure display. To relieve this slight pressure, press the toggle valve's handle down against the cabinet. This will slightly open the valve seat and relieve the pressure without building up new pressure when it is released.

Needle Valves

Needle valves are used to control the *rate* at which gas flows in and out of the unit.

The "GAS FLOW IN" RATE needle valve is used to control the flow rate into the MULTI-PYCNOMETER. It determines both the rate at which pressure builds in the Reference volume during a measurement (see Section IV) and – in combination with the VENT needle valve - the rate at which the sample is purged of contaminants (see Section III). *This valve should not be used to stop flow*. Flow of gas in to the unit is stopped using the "GAS FLOW IN" ON/OFF toggle valve.

The "VENT" needle valve controls the rate at which pressure is released when venting the sample cell (and reference volume) or when vacuum is required to decontaminate the sample (see Section III). To prevent elutriation of a powder sample as the pressure is released, open the Vent toggle valve and needle valve carefully to release pressure slowly. *This valve should not be used to stop flow*. Flow of gas out of the unit is stopped using the "VENT" OPEN/CLOSE toggle valve.

Selector Valve

This valve is used to expand gas from the reference volume, V_R, into the sample cell holder, V_C. It rotates 90degrees between "2 o'clock" (right) and "10 o'clock" (left); when pointing to the right only the reference volume (V_R) is in the gas path and when pointing to the left the sample cell holder is also included in the gas path ($V_C + V_R$).

Pressure Display and Transducer

Pressures within V_R and $V_R + V_C$ are displayed on the LCD in psig. (Note, since pycnometric measurements use pressure *ratios*, the units are in fact unimportant for calculating volume and density (but can be useful for setting the gas supply regulator and for troubleshooting). The display is zeroed using the zero knob to the right of the display when V_R is open to ambient (Gas flow toggle valve is OFF and vent toggle valve and needle valve are open).

When not used for prolonged times, the pressure transducer can exhibit some drift during the initial start up operation. To avoid this problem, it is recommended that after switching the unit on, the transducer undergo several pressurization/depressurization cycles prior to making an actual density measurement (or calibration).

Pressure Relief Valve (Internal)

A 25 psig (~172 kPa) pressure relief valve is located inside the unit immediately after the gas input fitting (See Figure 1). It is designed to prevent damage to the pressure transducer due to overpressurization. Gas cylinder regulator output pressures over 25 psig (~172 kPa) will activate the valve to vent excess gas pressure, in which case a hissing sound may be heard, and will prematurely empty your cylinder. Therefore the cylinder regulator should be set at only slightly more than 20 psig (138 kPa).

Calibration Status

The MULTIPYCNOMETER was calibrated at the factory and should only require recalibration after a sample cell or adapter sleeve has been replaced (for example if lost or damaged), after service or component replacement, if its new environmental temperature is outside the range 21-25°C, when required by your local regulations, and is recommended when the environmental temperature has changed by more than 4°C since the last calibration.

RS232 Port

Models with the D160E designation have an RS232 connection port on the left side of the unit, and are supplied with an RS232 cable and a software program ("PycData") on a CD which also contains an electronic copy of its own User Manual. PycData reads the pressure display and can perform all required calculations.

III. SAMPLE PREPARATION

Switch on the power to the unit using the mains on/off switch. Allow at least 15 minutes for the pressure transducer to warm up and stabilize, during which time you can prepare your first sample.

Purging Method

To purge contaminating vapors and atmospheric gases from the sample (and the system) attach a length of flexible plastic tubing to the hose barb connector on the right side of the MULTIPYCNOMETER and immerse the other end of the tubing into a beaker of water. Then perform the following steps.

1. Weigh accurately the clean, dry sample cell (cup). Record its (tare) weight, W_1.
2. Fill the sample cell (cup) to just below the slot/hole with dry sample. If the sample has been heated, ensure it has been cooled to room temperature (preferably in a desiccator) before proceeding. Record the total weight of cell plus sample, W_2.
3. Insert the cell with sample into the cell holder and replace the cover.
4. Close the "GAS FLOW IN" ON/OFF toggle valve and open the "VENT" toggle valve. Turn the selector valve to "CELL".
5. Open the "VENT" RATE needle valve fully counter-clockwise.
6. Turn the "GAS FLOW IN" RATE needle valve fully clockwise. *Do not over tighten it*. Open the "GAS FLOW IN" ON/OFF toggle valve.
7. Adjust the "GAS FLOW IN" RATE needle valve to give a slow rate of bubbling (1-2 bubbles per second) in the beaker of water then ***remove the tubing from the water.***
8. After 5-10 minutes of flow, close the "GAS FLOW IN" ON/OFF toggle valve.
9. See Section IV for analyzing the sample (volume measurement).

Vacuum Method

If you prefer to decontaminate your sample with vacuum, use the following procedure.

1. Close the "GAS FLOW IN" ON/OFF toggle valve and open the "VENT" toggle valve. Turn the Selector Valve to "CELL". Gently close the "VENT RATE" fully clockwise*; do not over tighten it.*
2. Attach vacuum tubing between the hose-barb connection on the right side and a vacuum pump. Switch on the pump.
3. Very slowly open the "VENT" RATE needle valve. If it is opened too rapidly, powder can be pulled out of the cell holder. Wait at least 10 minutes for the system to be evacuated.
4. To repressurize the system, close the "VENT" toggle valve, switch off the pump and remove the vacuum tubing. Open the "GAS FLOW IN" toggle valve, then slowly open the "GAS FLOW IN" RATE needle valve to admit analysis gas until the system is returned to ambient pressure.

IV. SAMPLE ANALYSIS

Stepwise measurement instructions

1. Ensure that the unit has been switched on for at least 15 minutes for the pressure transducer to warm up and stabilize.
2. Select the appropriate REFERENCE VOLUME for the size of sample cell by using the toggle valves “I” and “II” *according to the table printed on the unit* (below the zero knob).
3. Open the "VENT" toggle and "VENT” RATE needle valves. Wait for a stable near-zero reading.
4. Close the "VENT" toggle valve and set the display to zero.[1]
5. Turn the Selector valve to “V_R” (REF).
6. Open the “GAS FLOW IN” toggle valve, and pressurize to approximately 17 (psig) using the "GAS FLOW IN” RATE needle valve to control the rate of pressurization. Stop the flow by closing the “GAS FLOW IN” toggle valve.
7. Record the display reading after it has stabilized. This value is "P_1" in Equation (8).
8. Turn the selector valve to $V_C + V_R$ (CELL).
9. Record the display reading after it has stabilized. This value is "P_2" in Equation (8).
10. Vent the pressure slowly to prevent blowing powder out of the cell, by opening the "VENT" toggle valve with the "VENT” RATE needle valve slightly open.
11. Use equation (8) to calculate the true sample volume and density (see Worksheet).
12. Repeat steps 4 – 11 to obtain at least three replicate measurements. Average the results.

NOTE: The pressure transducer used in this pycnometer dissipates a very slight amount of heat. Because of its extreme sensitivity it can track the slight pressure increases associated with the heating of the gas. Accordingly, it is necessary to take the first reading observed after the digital display stabilizes. A change of approximately 0.001 on the digital display every 10-20 seconds is indicative of pressure increases due to heat dissipation and is normal. By taking the first reading after the display stabilizes and then rotating the selector valve and again immediately obtaining the first stable reading, the most accurate and rapid results will be achieved.

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1 If unable to zero the display please email qc.service@quantachrome.com for assistance.

MULTIPYCNOMETER WORKSHEET
TRUE DENSITY REPORT

SAMPLE I.D. _____ DATE _____
COMMENTS _____ OPERATOR _____

PREPARATION CONDITIONS _____

TOTAL WEIGHT (W_2) _____ g REFERENCE VOLUME (V_R) _____cm^3

TARE WEIGHT (W_1) _____ g CELL HOLDERVOLUME (V_C) _____cm^3

SAMPLE WEIGHT (W_2-W_1) _____ g

CALCULATE SAMPLE VOLUME:

$$V_S = V_C - V_R\left(\left(P_1 / P_2\right) - 1\right)$$

V_S = Volume of sample (cm^3)
V_C = Volume of Sample cell holder (cm^3)
V_R = Reference Volume (cm^3)
P_1 = Pressure reading after pressurizing just the Reference Volume
P_2 = Pressure reading after expanding gas into the Sample cell holder

CALCULATE SAMPLE DENSITY:

$$\text{Density} = (W_2\text{-}W_1) / V_S$$

DATA

	RUN 1	RUN 2	RUN 3	
P_1	_____	_____	_____	
P_2	_____	_____	_____	Average
V_S	_____	_____	_____	_____
DENSITY	_____	_____	_____	_____

V. CALIBRATION

Page 6 of this manual contains calibration information for reference during calibration and analysis calculations. Print that page and enter the values as indicated on the Calibration Tag or Printout provided with your instrument. This includes the sample cell volumes (V_C), the reference volumes (V_R), the volume of the large calibration sphere (V_{cal}large) and the volume of the micro calibration spheres (V_{cal}micro) provided with the MULTIPYCNOMETER. If, for any reason it is suspected that the value of V_C or V_R has been altered then recalibration should be performed. Powder blowing out of the sample cell into the tubing or operation at temperatures substantially different that room temperature will require recalibration.

Recalibration

To calibrate the MULTIPYCNOMETER the following steps should be followed in order:

1. Place the large calibration sphere into the large cell. Insert the large cell into the cell holder, replace and secure the cap.
2. Open toggle valves I and II.
3. Turn the selector valve to "CELL".
4. Open the "VENT" toggle and "RATE" valves.
5. Open the “GAS FLOW IN” toggle valve and adjust its "RATE" valve until the display shows about 1 (psig).
6. Purge the MULTIPYCNOMETER" in this mode for about 5 minutes.
7. Close the “GAS FLOW IN” toggle valve.
8. When the display shows a stable reading, set it to 0 using the zero knob and turn the selector valve to "REF".
9. Close the "VENT" toggle valve.
10. Open the “GAS FLOW IN” toggle valve until the pressure is approximately 17 (psi), then close the “GAS FLOW IN” toggle valve.
11. When the display is stable, note the pressure reading (P_1).
12. Turn the selector valve to "CELL".
13. When the display is again stable, note the new pressure reading (P_2).
14. Vent the reference volume and sample cell holder ($V_R + V_C$) by opening the "VENT" toggle valve.
15. Remove the calibration sphere from the large cell and repeat steps 1-14 noting the pressures P_1', P_2' with the selector valve in "REF" and "CELL" positions respectively.

16. Calculate the volume of the large Reference Volume ($V_R$large) using equation (9).

$$V_R large = \frac{V_{cal} large}{\left[\left(P_1' large / P_2' large\right) - 1\right] - \left[\left(P_1 large / P_2 large\right) - 1\right]} \quad (9)$$

Where:

V_{cal}*large* = volume of the *large* calibration sphere

P'_1 *large* = pressure in V_R *large* with *no* sphere in the cell.

P'_2 *large* = pressure in V_R *large* + V_C *large* with *no* sphere in the cell.

P_1 *large* = pressure in V_R *large* with the calibration sphere in the cell.

P_2 *large* = pressure in V_R *large* + V_C *large* with the sphere in the cell.

17. After solving equation (9) for V_R large use this value in equation (10) to calculate V_C large for the large sample cell.

$$V_C large = V_{cal} large + V_R large \left(\left(P_1 large / P_2 large\right) - 1\right) \quad (10)$$

18. Close toggle valve I and repeat steps 8-14.

19. Calculate the small reference volume using equation (11).

$$V_R small = \frac{V_C large}{\left[\left(P_1' small / P_2'^{*} small\right) - 1\right]} \quad (11)$$

Where:

P_1' *small* = pressure in V_R *small* with *no* sphere in the cell.

$P_2'^{*}$ *small* = pressure in V_R *small* + V_C *large* with *no* sphere in the cell.

20. Remove the large sample cell and insert the small adapter sleeve and small sample cell.

21. Repeat steps 3-14 and calculate the small cell volume using equation (12).

$$V_C\,small \;=\; V_R\,small\left[\left(P_1'small/P_2'small\right)-1\right] \tag{12}$$

Where:

P_1' *small* = pressure in V_R *small* with *no* sphere in the cell.

P_2' *small* = pressure in V_R *small* + V_C *small* with *no* sphere in the cell.

22. Remove the small sample cell and adapter and insert the micro adapter sleeve, micro sample cell and both micro calibration spheres.
23. Close toggle valves I and II.
24. Open the "GAS FLOW IN" toggle valve until the pressure is approximately 17 (psi). Then close the "GAS FLOW IN" toggle valve.
25. When the display is stable, note the pressure reading.
26. Turn the selector valve to "CELL".
27. When the display is again stable, note the new pressure reading.
28. Vent both the reference volume and cell holder by opening the "VENT" toggle valve.
29. Remove both calibration spheres from the micro cell and repeat steps 24-28 noting the pressures with the selector valve in "REF" and "CELL" positions.
30. Calculate the volume of the micro reference volume (V_R micro) using equation (13).

$$V_R micro = \frac{V_{cal}micro}{\left[\left(P_1'micro/P_2'micro\right)-1\right]-\left[\left(P_1micro/P_2micro\right)-1\right]} \tag{13}$$

Where:

V_{cal} *micro*= combined volume of both micro calibration spheres

P'_1 *micro* = pressure in V_R *micro* with *no* spheres in the cell

P'_2 *micro* = pressure in V_R *micro* + V_C *micro* with *no* spheres in the cell.

P_1 *micro* = pressure in V_R *micro* with the calibration spheres in the cell.

P_2 *micro* = pressure in V_R *micro* + V_C *micro* with the spheres in the cell.

31. After solving equation (13) for V_R micro use this value in equation (14) to calculate V_c micro for the micro sample cell.

$$V_C\ micro = V_{cal}\ micro + V_R\ micro\ \left(\left(P_1\ micro / P_2\ micro \right) - 1 \right) \qquad 14)$$

NOTE: If only the large sample cell is to be used, it is not necessary to recalibrate the small and micro cells. Similarly, if only the small cell is to be used, it is not necessary to calibrate the micro cell.

If the sample is analyzed at a temperature different by more than 4°C from that used to calibrate the instrument, the MULTIPYCNOMETER should be recalibrated at the new temperature.

Calibration Spheres

The table below shows the various spheres that are available for calibrating the instrument.

PART NUMBER	SIZE	DIAMETER (mm)	VOLUME (cm^3)
01500-MICRO[1]	Micro[2]	12.7	1.0772
01500-SMALL	Small	23.812	7.0699
01500-MEDIUM	Medium	38.1	28.958
01500-LARGE	Large[2]	47.625	56.5592

NOTE: The large and small calibration spheres are available with NIST measured diameters. Contact qc.sales@quantachrome.com for more details.

1. Sold in pairs.
2. Provided with the Multipycnometer.

VI. SOURCES OF ERROR

Non-ideal gas behavior

Equation (8) was derived using the equation of state for an ideal gas; therefore dry helium is recommended for use in the MULTIPYCNOMETER. However, dry nitrogen can also be used at room temperature often with no adverse effect. The use of gases which do not behave in a near ideal fashion at room temperature should be avoided.

Diffusion/Absorption

When analyzing vegetable matter, materials containing cellulose or low density polymers (including foams) it is preferred that nitrogen (or sulfur hexafluoride, SF_6) be used instead of helium because helium can diffuse into the solid matter and across cell walls.

Impure gases

If air or other gases which contain adsorbable impurities are used, the pressure readings will be affected due to adsorption on the sample surface. The extent of the resulting error depends upon the amount and nature of the impurities as well as the solid's surface area. Always use high purity gases and clean, metal gas lines – never plastic gas tubing.

Insufficiently prepared samples

Many samples contain impurities, usually adsorbed moisture, on their surface and within pores that should be removed prior to analysis. The presence of these impurities can affect the results in several ways:

1. The actual weight of the sample is less that the weight measured.
2. Contaminants fill pores causing a larger sample volume to be determined.
3. Volatile impurities will cause erroneous readings.

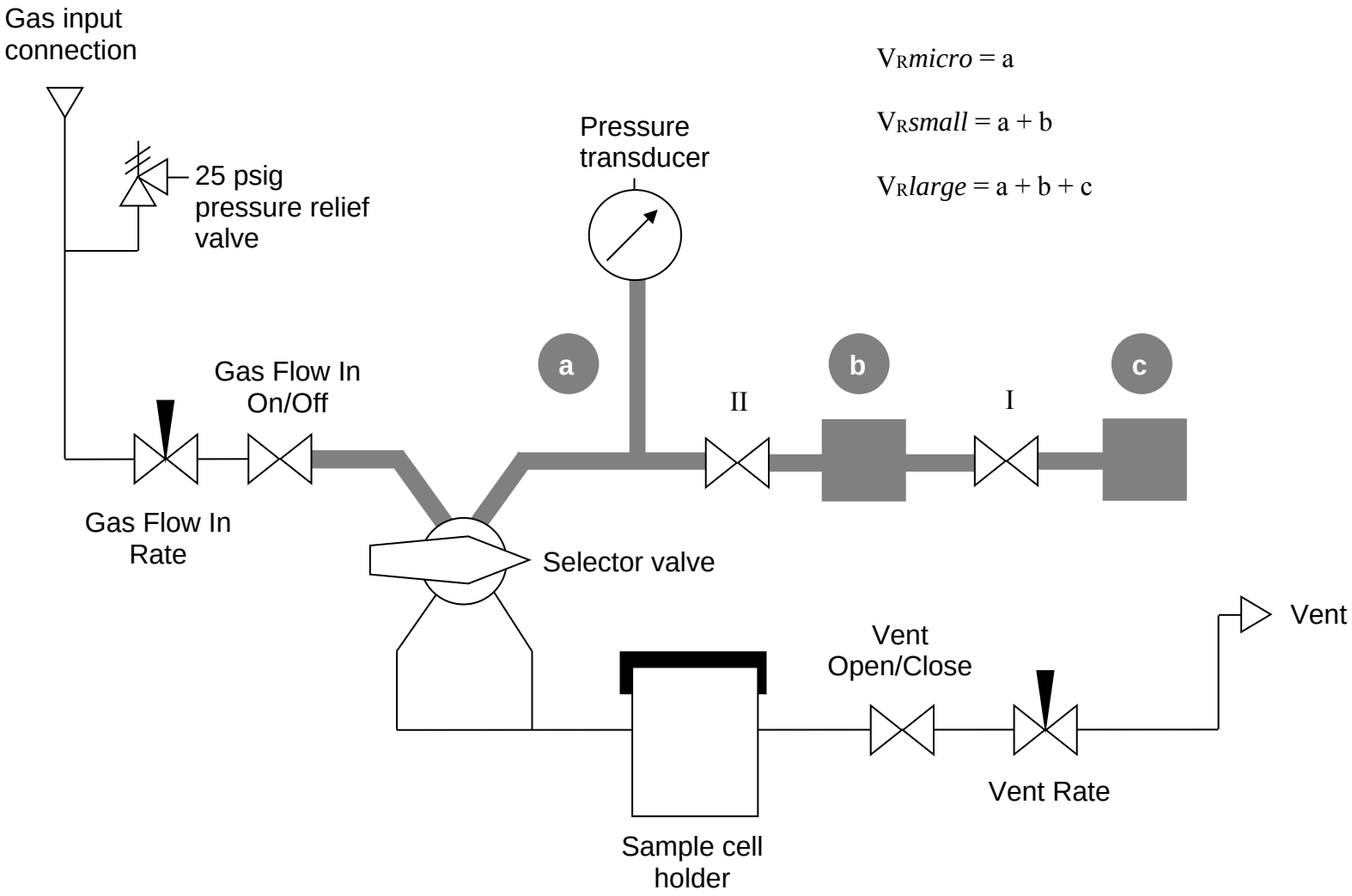
Successive volume determinations yielding results trending in one direction are usually an indication that contaminants are being removed after each cycle. Measurements should be repeated until two or three successive determinations are obtained to within 0.2%.

Another indication of the presence of volatile contaminants is a gradual pressure increase when the sample is included in the flow path (selector valve in the CELL position, $V_R + V_C$) after purging with dry gas. This occurs as the contaminants leave the surface and establish their own partial pressure.

The size of the gas molecule

An additional source of error in high surface area powders can be the annulus volume created between the powder surface and the center of mass of the gas phase molecules at the interface. Assuming that the closest approach of the center of mass of the gas molecules to the powder surface is 0.5Å (5×10^{-11} meter) and that the powder surface is in the order of 1000 square meters per gram, there will exist an annulus volume of 5×10^{-8} cubic meters (5×10^{-2} cm^3) per gram of powder. Thus, with samples of about 1 gram of high specific surface area, volume errors of 0.05 cm^3 can occur. Corrections for this error can be made with knowledge of the effective diameter (i.e., Van der Waals diameter) of the gas molecules and the powder's specific surface area.

Figure 1.


Gas input connection
25 psig pressure relief valve
Pressure transducer
$V_R micro = a$
$V_R small = a + b$
$V_R large = a + b + c$
a
b
c
II
I
Gas Flow In On/Off
Gas Flow In Rate
Selector valve
Vent
Vent Open/Close
Vent Rate
Sample cell holder